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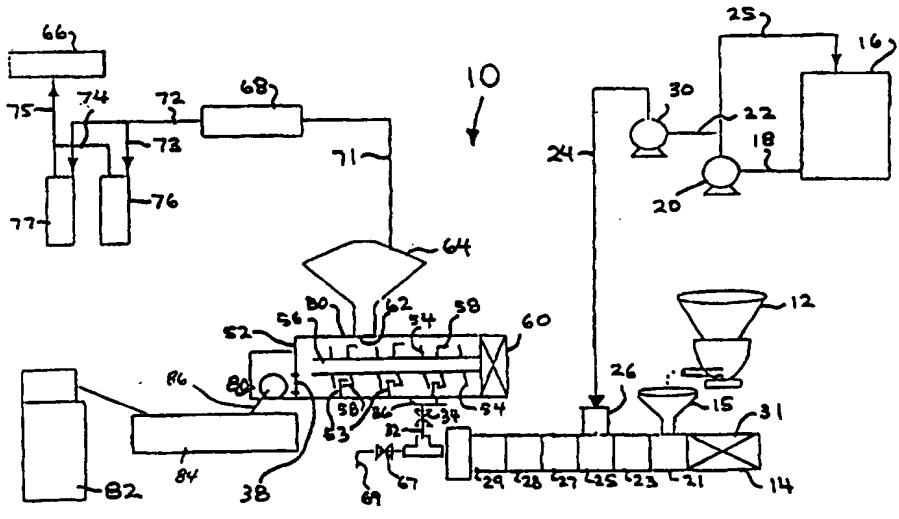
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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification ⁶ : C08F 6/10, B01D 1/22</p>	<p>A2</p>	<p>(11) International Publication Number: WO 98/02467 (43) International Publication Date: 22 January 1998 (22.01.98)</p>
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(54) Title: POLYMER RECOVERY



(57) Abstract

Methods for devolatilizing polymer solutions have been invented which include, in certain aspects, dissolving a viscous polymer in a solvent forming a polymer-solvent solution, introducing the polymer-solvent solution into a thermal dryer, heating or cooling the polymer-solvent solution in the thermal dryer forming product polymer with solvent removed and separated solvent (which may include other residuals), the separated solvent with other residuals if present vaporizing in the thermal dryer forming a vapor, removing the vapor from the thermal dryer, and discharging product polymer from the thermal dryer.

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POLYMER RECOVERY

This invention is directed to polymer devolatilization and recovery and, in certain aspects, to production of a polymer product with a residuals level of less than one thousand parts per million (ppm), the polymer product being produced from an initial polymer solution having a residuals level of up to fifty percent by weight, based on polymer weight. One particular method according to this invention is directed to the devolatilization of ethylene propylene diene monomer terpolymer (EPDM).

The prior art discloses a wide variety of polymer devolatilization and recovery systems and methods. Certain prior art methods have one or more disadvantages: a multi-stage process including a finishing step; required stripping of contaminants, residual solvent, and monomers; use of an immiscible fluid mixed with a flowing polymer to maintain heat transfer coefficient, increase diffusion driving force, or lower partial pressure; entrainment of polymer in overhead venting systems, known as "snowing", and polymer product flowing into venting systems, known as "vent flooding"; production of polymer product with inconsistent properties, such as a varying viscosity, or production of degraded product polymer or gels; and additional process unit operations to remove immiscible liquid or gas.

There has long been a need for an efficient, effective, single stage method for devolatilizing viscous polymers. There has long been a need for such a method which is relatively less expensive than existing methods and requires reduced capital outlay compared to existing methods. There has long been a need for such a method which produces a quality product with consistent properties without unacceptable viscosity variance, and without product degradation. There has long been a need for such a method which does not require additional stripping, an additional finishing step, or use of an immiscible fluid to maintain heat transfer. There has long been a need for such a method

5 in which snowing and vent flooding are significantly reduced and thereby improving product quality.

The present invention discloses systems and methods for polymer devolatilization. Devolatilization is the removal from a polymer product of unreacted monomer, solvent,
10 oligomers, and volatile condensation products, referred to herein collectively as "residuals".

In one aspect, the subject invention is directed to a method characterized by:

(A) introducing a polymer-solvent solution into a
15 thermal dryer,

(B) treating the polymer-solvent solution in the thermal dryer forming product polymer and separated solvent, separated solvent with residuals therein vaporizing in the thermal dryer forming a vapor
20 containing solvent and residuals,

(C) removing the vapor from the thermal dryer, and

(D) discharging polymer product having a solvent level of not more than 0.5 percent by weight solvent and other residuals from the thermal dryer.

25 In another aspect, a method according to the present invention includes: conveying a viscous polymer in solution with a solvent into a treatment vessel of a thermal dryer for devolatilization; applying a reduced pressure or vacuum within the treatment vessel; removing at least a portion of residuals
30 contained in the viscous polymer solution by way of the vacuum within the treatment vessel; and flowing polymer product from the treatment vessel for further processing, such as pelletizing.

In another aspect the method includes a step of cooling
35 or heating the viscous polymer solution in the treatment vessel to maintain polymer at desired temperature to achieve low residuals with acceptable product quality. In one aspect, such a method is oxygen-free.

40 Methods according to this invention may be either batch or continuous and may be used to recover any known viscous polymer. Methods according to this invention may be hydrous, or anhydrous (that is, in the absence of water), and an

5 optional volatile stripping agent may be used. Use of an immiscible heat transfer fluid is optional.

In certain embodiments, the viscous polymer solution is produced using a dissolver extruder that is fed pelletized polymer by a volumetric feeder. Solvent is injected into the
10 dissolver extruder under high pressure (for example 2000 pounds/square inch gauge (psig (13.8 MPa)) by a high pressure injection system. With such a system, solvent under pressure is injected into a polymer-filled zone in the dissolver extruder.

15 In another embodiment, the viscous polymer solution is produced in one or more solution polymerization reactors, operated either in parallel or in series. One such system is described in commonly owned United States Patent 3,914,342 (Mitchell). Preferably, the viscous polymer solution is
20 conveyed from the reactor(s) to the thermal dryer via single or multiple flash vessels to reduce solvent content.

A useful method to simulate this polymer recovery process is a dissolver extruder having six barrel sections through which polymer flows. Five of the six barrel sections are
25 heated by a fluid media, for example hot oil, pumping system. In certain preferred embodiments, the first of the six barrel sections is cooled, for example to about 30 degrees C (°C), to prevent polymer bridging at a polymer inlet of the dissolver extruder. A chilled glycol system provides sufficient cooling.

30 An electric, pneumatic, or hydraulic power system is used, in certain embodiments, to drive a rotating shaft in the dryer's treatment vessel to facilitate polymer drying and movement through the vessel. In one such aspect, the shaft is directly driven by an electric motor with a gear reducer. The
35 shaft is desirably sealed from exterior environments with a double mechanical seal with an inert buffer fluid (such as an oil buffer). In an additional aspect, a discharge end of the shaft also penetrates an exterior shell and is sealed with a double mechanical seal with an inert buffer (such as an oil
40 buffer) and is supported by one or more external bearings.

One vacuum system useful with methods according to the present invention has an oil-sealed vacuum pump and a

5 condenser (such as a shell and tube heat exchanger) disposed between the pump and a vent outlet of the treatment vessel. In one aspect, dual vapor collection traps are used alternately. Appropriate gauges indicate vacuum levels at desired points in the system.

10 In one embodiment, a single screw discharge device is used to convey polymer from the thermal dryer to downstream processing equipment. Twin screw or gear pump discharge devices are also suitable.

Further processing of devolatilized polymer product
15 flowing from the treatment vessel may include cooling, drying, and packaging. One method includes running the polymer product through a water bath and then pelletizing it in a pelletizing machine (such as a machine commercially available from Cumberland Strand Chopper Co.).

20 Methods according to this invention, as shown by the data presented below, produce substantially improved and unexpected results as compared to various prior art methods.

In certain embodiments, the present invention discloses a method for devolatilizing a polymer-solvent solution, the
25 method including conveying the polymer-solvent solution into a thermal dryer or forming the solution therein by introducing polymer and solvent into the thermal dryer, treating the polymer-solvent solution in the thermal dryer to separate polymer product from solvent and residuals, vaporizing at
30 least a portion of the solvent (or solvent with residuals therein) in the thermal dryer thereby forming a vapor containing solvent (or solvent and other residuals), removing the vapor from the thermal dryer, and discharging polymer product having at most 0.5 percent total residuals (including
35 solvent) by weight from the thermal dryer. The polymer product has a solvent level that is desirably less than 2000 ppm, preferably less than 1600 ppm, more preferably less than 1000 ppm. The polymer product also has a residual monomer content (such as a diene) in the polymer product of less than
40 100 ppm, preferably less than 50 ppm, and especially less than 10 ppm. The discharged polymer product is suitable for processing by a pelletizing machine. The method thus includes

5 feeding the polymer product to a pelletizing machine, and
producing pelletized polymer product. The polymer has a
Mooney viscosity that is desirably greater than 20, preferably
greater than 50, more preferably 70 or greater and generally
can be as high as 250, preferably as high as 120; or a melt
10 index (I_2) (ASTM D-1238 Condition 190/2.16) of less than 1 g/10
minutes to as low as 0.001 g/10 minutes.

The polymer is desirably selected from the group
consisting of ethylene/propylene/diene terpolymers (EPDM),
heterogeneous polyethylene, homogeneous polyethylene, linear
15 polyethylene, low density polyethylene, polypropylene,
polyurethane, ethylene propylene rubbers, and polystyrene.
Residence time of the polymer-solvent solution in the thermal
dryer is desirably less than 50 minutes, preferably less than
30 minutes and especially 15 minutes or less; Residuals
20 initially present in the polymer-solvent solution are
typically at a level of between 5 percent and 80 percent by
weight and are reduced by this method to a level in the
polymer product of less than 0.5 percent by weight;
preferably less than 0.2 percent by weight. The residuals
25 initially present in the polymer-solvent solution at a level
of 10 percent to 50 percent by weight are preferably reduced
to a level of less than 1000 ppm, or when residuals are
present initially at a level of 2-25 percent are reduced to a
level of 500 ppm or less, in the product polymer. The solvent
30 is typically a C_5 hydrocarbon or heavier, typically up to a C_{10}
hydrocarbon, or mixture of such hydrocarbons.

The method may also be performed without the addition of
oxygen, water or both. The method includes a variation
wherein polymer and solvent are continuously fed to the
35 thermal dryer and polymer product is continuously produced by
and conveyed from the thermal dryer. The method may be a
batch method.

Polymer product discharged from the thermal dryer is
desirably received by a discharge apparatus which conveys the
40 polymer product from the thermal dryer. The discharge
apparatus desirably is a discharge system with a housing, a
single screw conveyor rotatably mounted therein, and a direct

5 drive motor for rotating the single screw conveyor. The single
screw conveyor is preferably mounted in bearings and sealed at
its drive end with double mechanical seals to isolate
components from the housing from external influences such as
atmospheric gases, particularly oxygen. The housing has an
10 inlet and an outlet and the system desirably runs under
vacuum, for example 10-200 mm Hg (1.3-26.7kPa), preferably
about 25 mm Hg (3.3 kPa).

The method also includes heating or cooling the polymer-
solvent solution in the thermal dryer using dryer temperatures
15 maintained between 50°C and 290°C, preferably between 125°C
and 290°C, more preferably between 150°C and 220°C.

The method also may residual removing vapor by a vacuum
system that is in fluid communication with the thermal dryer.
The method from then includes condensing and collecting
20 solvent from the vapor removed by the vacuum system. The
method further comprises injecting nitrogen into the thermal
dryer ("nitrogen sweeping"). The polymer product desirably has
a viscosity that is substantially the same as that of polymer
used to form the polymer-solvent solution.

25 It is, therefore, an object of at least certain preferred
embodiments of the present invention to provide:

New, useful, unique, efficient, nonobvious methods and
systems for devolatilizing polymers;

Such methods which reduce residuals in a polymer from an
30 initial 5 percent to 80 percent content by weight to less than
0.5 percent, especially less than 0.2 percent by weight; and
such methods which preferably reduce residuals in a polymer
from an initial 10 percent to 50 percent content by weight to
less than 1000 ppm; and such methods which more preferably
35 reduce residuals in a polymer from an initial 5-25 percent
solvent content by weight to less than 500 ppm.

Such methods which achieve an acceptable polymer
residuals level in a single stage device;

Such methods which require no stripping and no immiscible
40 fluids to maintain heat transfer and diffusion rate;

Such methods in which snowing and vent flooding are
reduced and, preferably, substantially reduced; and

5 Such methods in which product polymer quality is
consistent, and such method in which a variety of varying
molecular weight solvents may be used (for example, isobutane,
cyclohexane, or ISOPAR™ E (a trademark of and made by Exxon
Chemical which is typically a mixture of C₈-C₁₀ hydrocarbons)).

10 Certain embodiments of this invention are not limited to
any particular individual feature disclosed here, but include
combinations of them distinguished from the prior art in their
structures and functions. Features of the invention have been
broadly described so that the detailed descriptions that
15 follow may be better understood, and in order that the
contributions of this invention to the arts may be better
appreciated. There are, of course, additional aspects of the
invention described below and which may be included in the
subject matter of the claims to this invention. Those skilled
20 in the art who have the benefit of this invention, its
teachings, and suggestions will appreciate that the
conceptions of this disclosure may be used as a creative basis
for designing other structures, methods and systems for
carrying out and practicing the present invention. The claims
25 of this invention are to be read to include any legally
equivalent devices or methods which do not depart from the
spirit and scope of the present invention.

 The present invention recognizes and addresses the
previously-mentioned problems and long-felt needs and provides
30 a solution to those problems and a satisfactory meeting of
those needs in its various possible embodiments and
equivalents thereof. To one skilled in this art who has the
benefit of this invention's realizations, teachings,
disclosures, and suggestions, other purposes and advantages
35 will be appreciated from the following description of
preferred embodiments, given for the purpose of disclosure,
when taken in conjunction with the accompanying drawings. The
detail in these descriptions is not intended to thwart this
patent's object to claim this invention no matter how others
40 may later disguise it by variations in form or additions of
further improvements. Further, the term "solution" as used
herein may include a slurry.

5

A more particular description of embodiments of the invention briefly summarized above may be had by reference to the embodiments which are shown in the drawings which form a part of this specification. These drawings illustrate certain preferred embodiments and are not to be used to improperly
10 limit the scope of the invention which may have other equally effective or legally equivalent embodiments.

Fig. 1 is a schematic view of a system useful in studying the methods according to the present invention without the
15 need to use a polymer reaction process as a feed stream.

Fig. 2 is a partial schematic view in cross-section of the dissolver extruder of the system of Fig. 1.

Fig. 3 is a schematic view of part of the system of Fig. 1.

20 Fig. 4 is a side schematic view of the thermal dryer of the system of Fig. 1.

Fig. 5A is a schematic view of a thermal dryer and associated apparatus useful in methods according to the present invention. Fig. 5B is a side view of a conveyor shown
25 in Fig. 5A.

Fig. 6 is a schematic view of a second system useful in methods according to the present invention.

Figs. 7-16 present data for methods according to the present invention.

30 Referring now to Fig. 1, a system 10 according to the present invention has a volumetric feeder 12 for feeding pelletized polymer to an inlet funnel 15 of a dissolver extruder 14; a thermal dryer 50; a solvent tank 16; and a vacuum system 66.

35 Solvent is pumped from the solvent tank 16 via lines 18 and 22 by a pump 20 to a pump 30. Pump 30 pumps the solvent through line 24 to an injector system 26. Injector system 26 injects the solvent into a third zone or barrel 25 of dissolver extruder 14.

40 As shown in Figs. 2 and 3, shaft 35 of dissolver extruder 14 is rotated by a drive system 31 that includes an electric motor 41, a drive belt 43, a high torque slip clutch 45, a

5 drive pulley 47 and a gear system 49 that is interconnected
with shaft 35. A valve 51 controls flow in line 19. A valve
53 controls flow in line 22. A valve 55 controls flow in
line 24. A gauge 57 indicates pressure in line 24. An
adjustable relief valve 59 provides relief protection for pump
10 30 and is in communication with valve 55 via the lines 63 and
65. As shown in Fig. 3, valve 67 controls the flow of
polymer-solvent solution in sample line 69.

Hot oil from a hot oil source 81 flows via line 83 to the
barrel sections 23, 25, 27, 28 and 29 of dissolver extruder
15 14. Hot oil exits these sections via lines 85, each with its
own control valve 87, and exit line 89. Dissolver extruder 14
produces a flowing solution of viscous polymer and solvent
which is fed via line 32 to thermal dryer 50 (Fig 1). Valve
34 controls delivery of polymer solution to dryer 50.

20 Thermal dryer 50 (see Figs. 1 and 4) has a shell 52 and
an apparatus therein for heating the flowing solution of
polymer and solvent and for moving it from inlet 36 to outlet
38. Typically a rotating hollow shaft 56 with mixing and
conveying elements 54 and 58 working in combination with
25 counter hooks 59 on the interior of shell 52 kneads and moves
the polymer-solvent solution within shell 52. The shell,
rotating shaft, counter hooks, movement elements, and mixing
elements may all serve as thermal transfer members for heating
or cooling the polymer-solvent solution. Heat transfer media
30 (such as steam or hot oil) may be pumped through a channel or
channels in each of these members. As shown in Fig. 4, a hot
oil system 90 is used in one embodiment to flow heat transfer
media through shaft 56 and related members. A drive system 60
is interconnected with and rotates shaft 56 (Figs. 1 and 4).
35 A source of hydraulic fluid under pressure (not shown)
provides drive fluid to drive system 60.

Certain residuals freed from the polymer-solvent solution
(for example vaporized volatiles, vaporized solvent, unreacted
monomers, oligomers) rise within vessel 52 and flow through an
40 upper outlet 62. In one embodiment, these residuals flow into
a vent dome 64 (Figs. 1 and 4). A vacuum system 66 evacuates
the residuals from shell 52 via lines 71, 72, 73, 74 and 75.

5 Residuals are condensed in a condenser 68 and collected in trap 76 or trap 77. The remaining residuals are removed via line 75.

Polymer product exits shell 52 of thermal dryer 50 and is moved through cooling medium 84 via line 86 into a cutter apparatus 82 to packaging equipment (not shown) (Fig. 1).

Fig. 5A illustrates one embodiment of a thermal dryer 100 (like the dryer 50) and associated apparatuses and connections. Dryer 100 has a hollow housing 102 with a shaft 104 extending therethrough for agitating and moving a polymer-solvent solution flowing into housing 102 through an inlet 116 (like the inlet 36, Fig. 1) into housing 102. Shaft 104 is rotated by a direct drive motor 106 interconnected with a gear box 108. External thrust bearings 110 and external radial bearings 112 facilitate rotation of shaft 104. There is no direct exposure of the bearings with thermal bearing 110 or radial bearings 112 to a flowing heating medium or to flowing polymer on the drive end (left end Fig. 5A). Preferably, the interior of the system is sealed and under high vacuum (typically to withstand a pressure operating range from 50 psig (2.4 kPa) pressure to a negative pressure of 750 mm Hg (100kPa) absolute vacuum). At each end of housing 102 a shaft-housing interface is sealed with double mechanical seals 114. Polymer product flows through an outlet 118 via conduit 119 to a discharge device 130 (like discharge apparatus 39, Fig. 4) which moves the polymer product from thermal dryer 100 for further processing. Coupling 120 allows injection of heat transfer media, such as hot oil, into the rotating main shaft 104 of thermal dryer 100. A channel in shaft 104 extends only to inlet 116 so that the heating medium does not heat apparatus beyond inlet 116. As with the system of Fig. 1, a vacuum system may be used with thermal dryer 100 to remove residuals. Thermal dryers are commercially available from Krauss-Maffei Verfahrenstechnik GmbH and List AG.

A single screw discharge device 130 (Fig. 5A), as depicted in Fig. 5B, is used to convey polymer product from thermal dryer 100 to downstream equipment. Single screw device 130 comprises a housing 134, a direct drive motor 131,

5 bearings 132 (typically including a back-thrust bearing and a roller bearing) to support a single screw 135 and to absorb the thrust of the screw, a double mechanical seal 133 to isolate components disposed within housing 134 from atmospheric contamination and prevent atmospheric oxygen
10 intrusion. Polymer product enters device 130 via inlet 136, (connected to outlet 118 of Figure 5a via conduit 119), is conveyed by screw 135 to discharge 137, and flows through discharge flange 138.

Advantages of using single screw device 130 or like
15 device include less mechanical drive complexity; true screw integrity providing good sealing capability (that is, less run-out and an ability to use a double mechanical seal); lower shear enabling better quality polymer product (less shear induced degradation); more efficient pumping of polymer from
20 inlet to outlet (less slippage); and better temperature control for downstream handling (such as pelletization).

Polymers useful in this invention include but are not limited to EPDM, heterogeneous polyethylene, for example, LLDPE polymerized as described in United States Patent
25 4,076,698 (Andersen et al); homogeneous polyethylene, as described in United States Patent 3,645,992 (Elston); substantially linear polyethylene as described in United States Patent 5,272,236 or 5,278,272 (Lai et al.); low density polyethylene (LDPE), and other thermoplastics such as
30 polypropylene, including those thermoplastics made in a solution, slurry or high pressure polymerization processes. Other polymers and copolymers include SIS and SBS type polymers, PELLATHANE™ (a trademark of The Dow Chemical Company) polyurethane, ethylene-polypropylene rubbers (EPR s),
35 and polystyrene. The novel methods described and claimed herein are surprisingly useful for high molecular weight, high viscosity elastomeric polymers.

Fig. 6 shows one system 200 useful in a continuous process according to the present invention. Polymer solution
40 enters a devolatilization vessel 202 via line 210 and solvent flashes off through an outlet 220. Polymer solution pools into an inlet of gear pump 208 and flows from it to a thermal

5 dryer system 206 (like that of Figs. 1-5B) and additional solvent is removed as described above. One or more additional devolatilization reactors may be used between the reactor 202 and the thermal dryer 206. In one aspect, the polymer-solvent solution is 50 percent to 70 percent by weight polymer and, therefore, is 50 percent to 30 percent by weight residuals.

EXAMPLES

In certain methods according to the present invention, ethylene propylene diene monomer ("EPDM") pellets as follows are fed to the dissolver extruder. Nordel™ material contains Irganox™ 1076 (a hindered phenolic antioxidant made by Ciba Geigy) in varying concentrations ranging between 100 ppm and 200 ppm.

EPDM Type* (electrical grade)	Specific Gravity	Mooney Viscosity
Nordel™ 2722	0.87	25 ± 5
Nordel™ 3681	0.87	45 ± 6
Nordel™ 5892	0.87	58 ± 5

*Pellet size of 0.125 inches (0.3cm)

20 Nordel™ is a trademark of DuPont Dow Elastomers L.L.C.

The dissolver extruder is a Werner and Pfleiderer Co. Model ZSK-30, co-rotating, fully intermeshing, twin screw, 30 millimeter (mm) extruder with a length over diameter (L/D) ratio of 21:1 in the extruder configuration with the screws being just over 20:1. The extruder is equipped with a Werner and Pfleiderer Co. high pressure injector located in the third downstream barrel section and is powered by a class one, division two, 15 horsepower, 3600 revolutions per minute alternating current motor, controlled by a digital speed controller. The extruder is starve fed resin by a K-Tron twin auger volumetric feeder, (with one of the two augers removed). The feed hopper on the feeder is loaded by hand and has a capacity of 10-20 pounds (3.7 -7.5kg). With one of the screws removed the feeder had a feed rate of 0-100 lb/hr (0-45kg/hr).

35 In examples described below, the solvent used is Isopar™ E solvent (made by Exxon Chemical) and in one aspect Isopar™ E solvent mixed with n-nonane, a C₉ hydrocarbon, at a level of

5 two percent by weight (to simulate residual diene since it has a higher boiling point than the Isopar™ E solvent).

The thermal dryer used in processing the three types of EPDM has a horizontal shell and a rotating shaft as shown in Fig. 1. Heat transfer media is pumped through the shell shaft and disc elements. The discs have mixing bars on their tips for clearing shell interior surfaces of product accumulation. Stationary counter hooks (see hooks 59, Fig. 4) are secured to the shell's interior and are shaped to interact with the rotating parts producing a mixing and kneading action. The discs are placed on the shaft at an angle to provide forward momentum and consistent product discharge. The dryer has a total volume of about 17 liters. A discharge apparatus 39 of the dryer is a twin screw extruder (having a Length-to-Diameter ratio of 7) powered by an hydraulic drive. The discharge section of the shell is attached to the middle section to give the discharge twin screw a horizontal position. As polymer is pushed forward by discs on the shaft, it is picked up by the twin screw and extruded through die holes located in an end flange.

Heat to the dryer is provided by a 40 watt exterior hot oil system. The hot oil system has its own pump and temperature controller providing hot oil to the thermal dryer at a desired temperature set point (for example 150-220°C). Hot oil is piped through a rotary union to the shaft (a hollow tube) and disc elements. The thermal dryer is made of three sections that are bolted together. Each of the three sections is jacketed and heat is provided by hot oil on the jacket side of each of these sections. The vent dome, (attached to the top of the unit by a flange connection), is channeled and hot oil flows through these channels to keep the dome hot.

The hydraulic drive system comprises two separate hydraulic units. One unit powers the main drive of the dryer which turns the shaft and the other unit powers the small twin screw drive. Both units have a rectangular shaped closed oil reservoir with high pressure oil pumps powered by alternating current motors mounted on the tops of the reservoirs. Maximum pressure of the units is adjustable with an internal relief

5 valve. Hydraulic oil is discharged from the pump through a
hand adjustable flow valve. High pressure hydraulic lines are
attached to the units and power drives using hydraulic line
quick disconnects. Hydraulic lines return from the drives
back to oil reservoirs. Pressure gauges on the hydraulic
10 drive units are used to estimate torque on the agitator and
twin screw extruder. By adjusting the position of the
discharge valve on one unit, the flow rate and thus the speed
of the drive to which it is connected are adjustable. Maximum
pressures at the agitator shaft are 150 bar (15 MPa).

15 An oil sealed vacuum pump is used for the vacuum system.
A shell and tube heat exchanger is used as a condenser between
the vent dome and the vacuum system, with chilled glycol on
the shell side at 1-5°C. Condensed vapors are collected in
one of two condensate traps (10 gallon (38 L) stainless steel
20 vessels) placed on weigh scales to measure rate. When one
trap is full, the other trap is valved in while the first trap
is emptied to a solvent drum by padding the trap with
nitrogen. The vacuum level in the dryer unit and in the
vacuum lines is measured via a valved-in connection with a
25 mercury filled manometer. The traps are equipped with gauges
to measure vacuum level.

Initially, a flange with three holes approximately 1/8
inch (0.3cm) in diameter with one hole plugged is used as a
die for the twin screw on the dryer. Later one hole is
30 enlarged to 3/16 inch (0.5cm) and the other two are plugged.
The melt is extruded into a 12 foot (3.7m) long water bath,
and then pelletized with a Cumberland Strand Chopper.

BATCH METHOD

35 In a batch method according to the present invention, the
drive system, vacuum system and heating system are as
described above. The dryer is also used, but is shortened by
about fifty percent in length (one section only is used) and
the discharge extruder is replaced with an end plate. Samples
40 are prepared for this trial by placing about 1 kg of Isopar™ E
solvent in a bag along with about 1 kg of polymer. The
polymer then absorbs the solvent and equilibrates. The

5 samples range from a gelatinous consistency of 25 Mooney
viscosity to a semi-solid material of about 70 Mooney
viscosity. The dryer is loaded with the contents of such bags
and the material is processed as described above.

This batch method data and results are shown in Table I.
10 The data demonstrate substantially improved, unexpected
results. Certain material is devolatilized from a solvent
content of 50 percent by weight to less than 0.2 percent by
weight.

In Table I, Products No. 1470 and 2522 are Nordel™
15 materials; "Oil Temp C" is the temperature of the heat
transfer media in the thermal dryer; "Agitator RPM" is the
speed of the shaft in the thermal dryer; "Vacuum (mm Hg(kPa))"
is the vacuum level in the system; "Batch Time" is the
residence time (in minutes) in the thermal dryer; "N₂ Sweep"
20 is the amount of nitrogen injected into the thermal dryer in
standard cubic feet per hour (standard cubic meters per hour);
"Prod. Temp. °C" is the temperature of the output polymer in
degrees Celsius; "Mooney Viscosity" is the viscosity of the
output polymer; and "Volatiles ppm" is the volatiles
25 (residuals) level of the output polymer.

CONTINUOUS METHOD

Table II presents the run conditions of the dissolver
extruder for 24 samples in a continuous method according to
30 the present invention. Because of the free volume area of the
screws in the feed throat of the 30 mm twin screw extruder,
only samples of EPDM in 1/8 inch (0.3cm) pellet form were
used. On start up of the dissolver extruder, low feed rates
to the extruder of low Mooney viscosity EPDM (20
35 pounds/hour (7.5 kg/hr) Nordel™ 2722), are used to form an
initial melt seal within the extruder before starting any
solvent feed to the extruder. The dissolver extruder is run
at low rates without solvent until polymer flow is established
in the thermal dryer. High extruder discharge pressures (for
40 example greater than 1000 psi (7MPa)) is necessary to clear the
line (½ inch (1.3cm) on inside diameter) between the dissolver
extruder discharge and the dryer. Solvent is injected into

5 barrel zone three (barrel section 25, Fig. 1), through a high
pressure injector with a back pressure of 600-800 psig (4.1 -
5.5 MPa). Percent solvent-to-EPDM by weight is kept low (10-
20 percent) initially. When solvent is being fed to the
10 extruder, a detector at the feed throat is used to check for
solvent flashing back to the feed hopper. After establishing
solvent to the extruder with no problems, the percent solvent
to EPDM ratio is increased to a 50 weight percent level over a
three or four step process. As the relation of the solvent
percentage EPDM members, total motor torque decreases,
15 allowing much higher throughput rates than dry (that is,
containing no solvent) EPDM materials. Desired rates are
achieved by increasing polymer addition rates first and then
increasing solvent flow rates. Maximum solvent addition is
limited to 25-27 pounds/hour metric (9.3 -10 kg/hr) because
20 of pump (pump 30, Fig. 1) limitations. Samples 1-8 and 22-24
are Nordel™ 5892 material. Samples 9-15 are Nordel™ 2722
material. Samples 16-21 are Nordel™ 3681 material.

In Table II, "Polymer Mooney" is the viscosity of the
input polymer; "Polymer Feed" is the rate of input polymer
25 feed to the thermal dryer in pounds per hour (kg/hr); "Solvent
Feed" is the rate of solvent feed to the thermal dryer in
pounds per hour (kg/hr); "Res Time min" is the residence time
(calculated average) in minutes of the polymer-solvent
solution in the thermal dryer; "Torque Bar" is the drive motor
30 torque measured in bars (MPa); Other column headings have the
same meaning as in Table I.

The temperature of the 50 percent EPDM stream is 180 to
220°C . To control the discharge temperature of the dissolver
extruder at around 200°C , the screw speed is adjusted up or
35 down within a limited range. The maximum screw speed of the
dissolver extruder is 600 revolutions per minute, while the
lower limit is set by drive torque and/or high percent fill of
the screws. Partially closing the valve on the polymer line
going to the thermal dryer (that is increasing the extruder
40 discharge pressure) also increases discharge temperature.
Screw speed, discharge pressure, and control of the extruder
barrel zone temperatures (barrel zones 23, 25, 27, 28, 29),

5 are all used so that the polymer supply to the thermal dryer remains in the target temperature range.

The dissolver extruder runs at steady amp loads and steady discharge pressures, all indicative of good solvent incorporation. Samples of the solution are periodically taken
10 manually via discharge line 69 (Fig. 3) and physically inspected to ensure that a uniform solution is being generated.

Table II presents the run conditions of the 24 samples. The dryer is preheated to 150°C with the hot oil system
15 before being fed a polymer stream. When the polymer stream is introduced to the unit, the hydraulic shaft drive is started and the shaft speed adjusted to the desired number of revolutions per minute. When solvent is added to the polymer stream, the vacuum is valved to the thermal dryer and adjusted
20 to desired vacuum levels. Using sight glasses on top of the dryer, polymer level in the dryer is monitored. When the desired level is reached, the twin screw hydraulic drive unit is started up. By adjusting the speed of the twin screw, the amount of polymer being discharged from the unit can be
25 matched to the amount of polymer entering, and in this manner the level in the thermal dryer remains relatively constant.

Product consistency is characterized by Mooney viscosities. Fig. 7 shows the Mooney viscosity for the 24 samples on the left vertical axis and the sample number on the
30 horizontal axis. The three solid horizontal lines represent the starting Mooney viscosities for the EPDM materials. In all cases, unexpectedly, little or no Mooney drop is experienced. Residence times of the polymer in the dryer are as long as 41 minutes, with discharge melt temperatures as
35 high as 287°C. Shaft speeds in the dryer as high as 70 revolutions per minute are evaluated and torque loads as high as 139 bar metric (13.9 MPa) (as measured on the hydraulic drive unit) are observed. None of these conditions produce any significant polymer degradation. The Nordel™ EPDM
40 products contain standard antioxidant packages (for example, 1500 ppm Irganox™ 1076 (a hindered phenolic antioxidant made by Ciba-Geigy Corporation)).

5 Devolatilization performance is measured by headspace gas chromatography of the final products. This is accomplished by sampling the head-space of a sealed sample vial containing final product from the thermal dryer within a few seconds (that is, within less than 15 seconds) of discharge. For each
10 sample, a known amount of product (approximately 0.5 gram) is placed in a septum sealed vial. The sample vial is placed on an automated headspace analyzer connected to a gas chromatograph. The sample vial contents are then analyzed using a quantitative multiple headspace extraction procedure.
15 The concentration of the residual solvent in the sample vial is determined from the quantitative analysis of known solvent standards analyzed under identical multiple headspace extraction conditions.

Fig. 8 presents the residual levels (vertical axis
20 indicates parts per million (ppm) Isopar™ E solvent) for the 24 samples (sample number indicated on horizontal axis). Only one sample is above 2,000 ppm and it was run with a vacuum level of 200 mm Hg (27 kPa). Within the ranges of temperature, vacuum level, residence time and other variables,
25 19 samples unexpectedly fell below 1,000 ppm residual solvent.

Typically the devolatilization of EPDM materials with prior art extrusion equipment results in vent fouling due to snowing. EPDM rubbers with higher viscosities typically have a greater problem. In the vent dome of the dryer, only a
30 small amount of snow is present in the lower portion or section of the dome. Accumulation over several hours of running time is minor. This represents an unexpected result of the present invention.

Fig. 9 shows the relationship of vacuum level, residence
35 time, and the residual levels of devolatilized EPDM (Nordel™ 2722). Vacuum level in mm Hg absolute (with value in kPa being indicated in parentheses) is set forth on the left vertical axis, and is indicated as the first bar in each pair of bars for each sample. Residence time in minutes is also
40 set forth on the left vertical axis (the values which are not enclosed in parentheses, e.g., 50, 45, etc.), and is indicated as the second bar in each pair of bars for each sample.

5 Solvent residuals in parts per million is set forth on the
right vertical axis, and is indicated by the connected plotted
small boxes. Sample number is indicated on the horizontal
axis. For Fig. 9, the polymer contains from 25 to 50 percent
solvent upon entering the dryer, and the dryer is filled to 40
10 percent.

As set forth in Fig. 9, as vacuum level increases and or
residence time decreases, the residual solvent level in the
polymer increases. One sample, sample number 15, is not
included in data supporting Fig. 9 because its starting
15 percent solvent level in the EPDM is low (10 percent).

Figs. 10 and 11 also show the same relationship as Fig. 9
(with the axis and bars being defined as described above with
respect to Fig. 9), but with the other EPDM materials (Nordel™
3681 and Nordel™ 5892). Also, for Fig. 10, the polymer
20 contains from 40 to 50 percent solvent upon entering the dryer
and the dryer is filled to 25 to 40 percent, while, for Fig.
11, the polymer contains from 40 to 50 percent solvent and the
dryer is filled to 40 to 50 percent. The same trends can be
seen for these two EPDM's as for that of Fig. 9. In Fig. 11,
25 one sample is again left out (sample number 7) because of a
low starting solvent level (12 percent).

Table III presents information regarding certain samples
referred to in Figs. 9, 10, and 11. Certain variables are
varied for these samples, including how full the thermal dryer
is ("Fill percent"); whether nitrogen is present and, if so
30 how much, ("N₂ SCFH (m³/hr)"); rotational speed of the thermal
dryer shaft ("RPM"); the amount of material being fed to the
thermal dryer, polymer plus solvent, in pounds per hour
("Total Feed lb/hr"); the temperature of the heat transfer
35 media as it is fed to the thermal dryer, 150°C, or any
indication that it is not used ("Oil Temp (°C)"); and the
amount of solvent as a weight percent of the total feed to the
thermal dryer ("Solvent percent").

Samples 7 and 15 in Table II have starting solvent
40 levels in the range of 10 percent by weight. The two samples
are run under different conditions, but the devolatilized EPDM

5 final product, unexpectedly has residual levels of less than 100 ppm.

Two-thirds of the samples are run with a nitrogen sweep of the dryer at a level of 2 to 5 standard cubic feet per hour (0.06 to 0.14 m³/hr). The rest of the samples are run without
10 nitrogen. Table II presents three pair of samples, one of each of the three different EPDM materials, with and without nitrogen sweep. These sample pairs are run under the same conditions except for the nitrogen. The data indicates lower solvent residual levels for the Nordel™ 3681 and Nordel™ 5892
15 with nitrogen. The Nordel™ 2722 samples show statistically the same residual level.

Some of the samples are collected after adding about 2 percent n-nonane by weight to the Isopar™ E solvent. The samples are tested for n-nonane residuals and the data is
20 presented in Fig. 12. (The right vertical axis of Fig. 12 shows the weight percent of n-nonane in the total residuals). N-nonane is present in the total residuals in a 3-6.5 percent by weight range).

Three different temperature profiles are used for the
25 above described samples. First, the hot oil system runs at 200°C and the jacket side of the shell on all three sections of the dryer and the vent dome are valved in. Hot oil temperatures of the hot oil system have an average 5°C change (in versus out). Polymer temperatures range from 211°C-
30 233°C in the second section of the dryer. The second condition runs the hot oil system at 150°C with the jacket side of the shell on all three sections of the dryer and the vent dome valved in. Hot oil temperatures in and out of the hot oil system have a change of 3°C. Polymer temperatures range from
35 167-187°C at the same point. The third condition runs the hot oil system at 150°C with the vent dome valved in and all three sections of the unit valved out after the dryer is running. The polymer temperatures range from 115-205°C.

Fig. 13 presents data for the three different Nordel™
40 EPDM materials run at the same dryer fill level (40 percent), dryer shaft rotation speed (50 rpm), and approximately the same dryer residence time (26 minutes). Both the temperature

5 of the EPDM materials in the dryer and the peak recorded
hydraulic pressure required to turn the shaft at 50 RPM's are
shown. The vertical axis shows the torque measured in bar
(with the value in MPa being set forth in parentheses). The
vertical axis further shows the dryer polymer temperature in °C
10 (the value on the vertical axis which is not in parentheses,
that is 250, 200, 150, etc.). This data, unexpectedly, shows
no significant difference in torque requirements between the
27 Mooney, 50 Mooney, and the 65 Mooney EPDM materials. A
possible reason for the similarity in torque requirements for
15 the three different EPDM materials is a change in condition of
the polymer melt at the varying temperatures (from a
continuous melt to a discontinuous melt, that is, crumbs).
The average temperatures are: 27 Mooney viscosity material =
180°C, 50 Mooney viscosity material = 190°C, and 65 Mooney
20 viscosity material = 215°C. If these temperatures are
reached through a mechanical means, there would have been
higher torque requirements for the higher Mooney EPDM
materials.

Data regarding thermal dryer run conditions, presented in
25 Fig. 14-16, shows sample differences and torque requirement
similarities. In each of Fig. 14 - 16, the vertical axis
shows torque requirement in bar (with the value in MPa being
set forth in parentheses. In each of Fig. 14-16, the vertical
axis further shows disc temperature in °C (the value on the
30 vertical axis which is not in parentheses, that is 240, 220,
etc.)

In conclusion, therefore, it is seen that the present
invention and the embodiments disclosed herein and those
covered by the appended claims are well adapted to carry out
35 the objectives and obtain the ends set forth. Certain changes
can be made in the subject matter without departing from the
spirit and the scope of this invention. It is realized that
changes are possible within the scope of this invention and it
is further intended that each element or step recited in any
40 of the following claims is to be understood as referring to
all equivalent elements or steps.

Table 1

Trial No.	Product No.	Oil Temp °C	Agitator RPM	Vacuum mm Hg (kPa)	Batch Time min	N ₂ Sweep scfh (m ³ /hr)	Frcd. Temp. °C	Mooney Viscosity	Volatiles ppm
1	1470	200	25	35 (4.7)	30	None	196	74.4	1100
2	1470	200	50	35 (4.7)	30	None	215	87.4	3000
3	1470	200	50	35 (4.7)	15	None	198	71.4	3500
4	2522	200	25	35 (4.7)	30	None	191	21.9	3000
5	1470	200	25	35 (4.7)	30	5 (0.14)	200	64.2	40
6	1470	200	50	10 (1.3)	15	5 (0.14)	190	75.7	90
7	1470	200	50	10 (1.3)	15	None	194	72.2	920
8	1470	200	50	35 (4.7)	30	5 (0.14)	210	71.9/84.4*	< 20
9	1470	200	50	10 (1.3)	30	5 (0.14)	213	62.6	25
10	1470	200	50	35 (4.7)	30	None	206	68	3000
11	2522	200	50	35 (4.7)	30	None	204	22.2	2500
12	2522	150	50	35 (4.7)	30	None	162	21.9	160
13	unknown	150	50	35 (4.7)	15	None	160	CND**	1400
14	1470	200	25	35 (4.7)	30	None	198	62.9	400
15	1470	150	25	35 (4.7)	15	None	135	74.1	4200
16	2522	150	50	35 (4.7)	10	None	137	21.3	6500
17	1470	150	25	35 (4.7)	15	5 (0.14)	138	67.7	5900
18	unknown	150	50	35 (4.7)	30	None	159	None	None

*Two separate trial results reported here; **CND = could not determine

Table II

Trial No.	Polymer Mooney	Polymer Feed (lbs/hr)	Solvent Feed (kg/hr)	Oil Temp (°C)	Agitator RPM	Vacuum mm Hg (kPa)	Res. Time min	% Sweep scfh (m ³ /h)	Prod. Temp (°C)	Torque Bar (MPa)	Mooney Viscosity	Volatiles ppm
1	65	30 (11)	30 (11)	200	50	50 (6.7)	26	5 (0.14)	215	111 (11.1)	64.3	522
2	65	30 (11)	30 (11)	200	50	15 (2.0)	26	2 (0.056)	214	100 (10.0)	65.1	484
3	65	30 (11)	30 (11)	200	50	100 (13.3)	26	None	214	98 (9.8)	64.6	1403
4	65	30 (11)	30 (11)	200	50	50 (6.7)	26	None	222	111 (11.1)	65.3	817
5	65	40 (15)	30 (11)	200	50	50 (6.7)	26	5 (0.14)	218	136 (13.6)	64.7	657
6	65	30 (11)	20 (7.5)	200	50	50 (6.7)	26	5 (0.14)	219	104 (10.4)	65	486
7	65	20 (7.5)	2 (0.7)	200	50	15 (2.0)	41	5 (0.14)	233	118 (11.8)	67.5	<20
8	65	30 (11)	30 (11)	150	50	50 (6.7)	26	5 (0.14)	187	118 (11.9)	65.8	1159
9	25	30 (11)	30 (11)	150	50	50 (6.7)	26	5 (0.14)	167	80 (8.0)	28.2	1241
10	25	30 (11)	30 (11)	150	50	15 (2.0)	26	5 (0.14)	165	78 (7.8)	28.5	434
11	25	30 (11)	30 (11)	150	50	15 (2.0)	26	None	167	91 (9.1)	28.8	419
12	25	60 (22)	30 (11)	150	60	15 (2.0)	15	None	175	107 (10.7)	28.3	512
13	25	80 (30)	30 (11)	150	70	15 (2.0)	13	None	182	139 (13.9)	28.2	836
14	25	30 (11)	30 (11)	None	50	15 (2.0)	26	None	192	104 (10.4)	29.1	477
15	25	36 (13)	4 (1.5)	None	50	15 (2.0)	25	5 (0.14)	187	94 (9.4)	28	100
16	45	30 (11)	30 (11)	None	50	15 (2.0)	26	5 (0.14)	183	109 (10.9)	49	257
17	45	30 (11)	30 (11)	None	30	15 (2.0)	26	5 (0.14)	151	95 (9.5)	49.3	341
18	45	40 (15)	30 (11)	None	50	15 (2.0)	20	5 (0.14)	187	124 (12.4)	49.8	806
19	45	30 (11)	30 (11)	None	50	15 (2.0)	26	None	205	111 (11.1)	51.9	317
20	45	30 (11)	30 (11)	None	50	200 (26.6)	26	None	198	133 (13.3)	49.6	4712
21	45	30 (11)	30 (11)	None	50	15 (2.0)	14	5 (0.14)	149	58 (5.8)	50.8	941
22	65	30 (11)	30 (11)	None	50	15 (2.0)	14	5 (0.14)	128	48 (4.8)	65.4	948
23	65	30 (11)	30 (11)	None	30	15 (2.0)	33	5 (0.14)	134	111 (11.1)	65.4	1597
24	65	30 (11)	30 (11)	None	50	15 (2.0)	26	5 (0.14)	203	100 (10.0)	65.6	334

Table III

Figure	Sample	Vacuum mm Hg (kPa)	N ₂ scfh (m ³ /h)	Residence Time (min)	Fill (%)	RPM	Total Feed lb/hr (kg/hr)	Oil Temp (°C)	Solvent (%)
9	11	15 (2.0)	None	26	40	50	57 (21)	150	47
9	10	50 (6.7)	5 (0.14)	26	40	50	57 (21)	150	47
9	14	15 (2.0)	None	26	40	50	57 (21)	Oil off	48
9	12	15 (2.0)	None	16	40	50	87 (32)	150	31
9	13	15 (2.0)	None	13	40	50	107 (40)	150	25
9	9	50 (6.7)	5 (0.14)	26	40	70	57 (21)	150	47
10	16	15 (2.0)	5 (0.14)	26	40	50	57 (21)	None	48
10	19	15 (2.0)	None	26	40	50	57 (21)	None	47
10	17	15 (2.0)	5 (0.14)	26	40	30	57 (21)	None	48
10	18	15 (2.0)	5 (0.14)	20	40	50	67 (25)	None	41
10	21	15 (2.0)	5 (0.14)	14	25	50	57 (21)	None	47
11	24	15 (2.0)	5 (0.14)	26	40	50	57 (21)	None	47.4
11	2	15 (2.0)	2 (0.056)	26	40	50	56 (21)	None	47
11	6	50 (6.7)	5 (0.14)	26	40	50	48 (18)	None	38
11	1	50 (6.7)	5 (0.14)	26	40	50	55 (20)	None	46
11	5	50 (6.7)	5 (0.14)	26	40	50	66 (25)	None	39
11	4	50 (6.7)	None	26	40	50	55 (20)	None	46
11	22	15 (2.0)	5 (0.14)	14	25	50	59 (22)	None	49
11	8	15 (2.0)	5 (0.14)	26	40	60	57 (21)	None	47
11	3	100 (13.3)	None	26	40	50	57 (21)	None	47
11	23	15 (2.0)	5 (0.14)	33	50	30	57 (21)	None	47

Table IV*

MOONEY	inlet temp °C	vacuum mmHg (kPa)	shaft RPM	shaft TORQUE (l lbs (kg.m))	poly temp °C	Extruder RPM	Extruder discharge temp °C	Extruder discharge (MPa)	Volatiles ppm ENB	Volatiles ppm TOTAL
69.4	146.3	14.2 (1.89)	29.4	667.2 (92.24)	202.9	66.8	212.3	218.2 (1.504)	9.0	258.0
63.3	144.5	14.8 (1.97)	29.4	630.4 (87.16)	202.7	66.8	211.3	205.3 (1.415)	8.0	199.0
70.2	147.7	14.5 (1.93)	29.4	632.2 (87.41)	203.5	66.7	213.8	219.2 (1.511)	5.0	111.0
65.7	146.4	14.6 (1.95)	29.4	636.3 (87.97)	206.4	59.9	214.7	199.5 (1.376)	5.0	141.0
65.6	146.9	14.5 (1.93)	29.4	674.9 (93.31)	206.2	59.9	216.8	209.9 (1.447)	7.0	190.0
69.6	148.9	13.8 (1.84)	29.4	661.9 (91.51)	206.4	59.9	214.5	216.8 (1.495)	8.0	213.0
70.6	150.0	14.6 (1.95)	25.0	816.2 (112.8)	205.2	59.7	215.4	250.1 (1.724)	17.0	500.0
71.0	150.5	14.1 (1.88)	25.0	694.6 (96.03)	205.6	59.8	212.3	234.3 (1.615)	10.0	271.0
71.3	150.4	14.0 (1.87)	25.0	586.8 (81.13)	204.7	60.0	210.7	208.3 (1.436)	8.0	246.0
71.4	150.7	14.5 (1.93)	20.0	755.9 (104.5)	204.7	59.9	209.7	227.7 (1.570)	15.0	445.0
70.4	150.4	14.4 (1.92)	20.0	811.3 (112.2)	204.9	59.9	208.3	238.8 (1.646)	12.0	337.0
70.7	150.1	14.6 (1.95)	20.0	829.6 (114.7)	204.8	59.8	206.9	244.9 (1.689)	11.0	315.0
71.3	150.6	15.0 (2.00)	15.1	938.0 (129.7)	205.5	59.8	208.9	249.0 (1.717)	20.0	565.0
71.7	149.8	14.0 (1.87)	15.0	845.3 (116.9)	204.2	59.9	201.4	226.7 (1.563)	6.0	153.0
70.6	149.4	13.8 (1.84)	15.0	780.0 (107.8)	204.4	59.9	202.8	230.5 (1.589)	15.0	422.0

*Total residuals on the feed was 23-24 ounces (650-680 grams)

Table V

Sample Number	Dupont Norder EPDM	Disc Screw rpm	Disc Torque Bar	Total Feed lbs/hr (kg/hr)	Solvent in feed	Hot Oil to Disc Temp °C	Disc No. 1 Temp °C	Disc No. 2 Temp °C	Disc No. 3 Temp °C	Discharge Melt Temp °C	Aprox Fill Level (%)	Residence Time Minutes	Vacuum mm Hg (kPa)	N ₂ Sweep SCFH (m ³ /hr)	Mooney Viscosity	Isopar-E Volatiles ppm
10	2722	50	78 (7.8)	56.7 (21.2)	47.1	150	155	155	167	178	40	26	15 (2.0)	5 (0.14)	28.5	434
11	2722	50	91 (9.1)	57.4 (21.4)	47.7	150	154	154	167	182	40	26	15 (2.0)	none	28.8	419
15	2722	50	94 (9.4)	40.2 (15.0)	10.4	none	191	177	187	206	40	25	15 (2.0)	5 (0.14)	28.0	100
14	2722	50	104 (10.4)	57.7 (21.5)	48.0	none	156	162	192	190	40	26	15 (2.0)	none	29.1	477
16	3681	50	109 (10.9)	57.2 (21.3)	47.6	none	134	141	183	196	40	26	15 (2.0)	5 (0.14)	49.0	257
19	3681	50	111 (11.1)	57.8 (21.6)	48.1	none	132	147	205	230	40	26	15 (2.0)	none	51.9	317
18	3681	50	124 (12.4)	67.2 (25.1)	40.5	none	157	153	187	221	40	20	15 (2.0)	5 (0.14)	49.8	806
20	3681	50	133 (13.3)	57.0 (21.3)	47.4	none	140	144	198	225	40	26	200 (26.8)	none	49.6	4712
3	5892	50	98 (9.8)	56.7 (21.2)	47.1	200	187	211	157	250	40	26	100 (13.4)	none	64.6	1403
24	5892	50	100 (10.0)	57.0 (21.3)	47.4	none	35	134	137	287	40	26	15 (2.0)	5 (0.14)	65.6	334
2	5892	50	100 (10.0)	56.3 (21.0)	46.7	200	188	214	154	221	40	26	15 (2.0)	2 (0.056)	65.1	484
6	5892	50	104 (10.4)	48.2 (18.0)	37.8	200	196	219	159	230	40	26	50 (6.7)	5 (0.14)	65.0	486
1	5892	50	111 (11.1)	55.0 (20.5)	45.5	200	190	214	145	220	40	26	50 (6.7)	5 (0.14)	64.3	522
4	5892	50	111 (11.1)	55.4 (20.7)	45.8	200	190	222	140	241	40	26	50 (6.7)	none	65.3	817
5	5892	50	136 (13.6)	65.7 (24.5)	39.1	200	197	218	162	247	40	26	50 (6.7)	5 (0.14)	64.7	657

- 5 1. A method for devolatilizing a polymer-solvent solution,
characterized by a single iteration of the following
sequential steps:
- 10 (A) introducing a polymer-solvent solution having a
solvent plus residuals content of less than 80
weight percent into a thermal dryer,
- (B) treating the polymer-solvent solution in the thermal
dryer to form product polymer and separated solvent,
the separated solvent with residuals therein
15 vaporizing in the thermal dryer to form a vapor
containing solvent and residuals,
- (C) removing the vapor containing solvent and residuals
from the thermal dryer, and
- (D) discharging from the thermal dryer polymer product
20 having not more than 0.5 percent by weight solvent
and residuals.
2. The method of claim 1 wherein the polymer-solvent
solution is continuously fed to the thermal dryer and
polymer product is continuously produced by and conveyed
25 from the thermal dryer.
3. The method of claim 1 wherein product polymer discharged
from the thermal dryer is received by a discharge
apparatus which conveys the product polymer from the
30 thermal dryer, wherein the discharge device system is
characterized by:
- a single or twin screw conveyor rotatably mounted
in a housing, the housing having an interior into which
product polymer flows through a housing inlet,
- 35 a direct drive motor at a drive end of the
housing connected to the single or twin screw conveyor
for rotating it,
- dual mechanical seals at the drive end of the
housing for sealing an interface of the single or twin
40 screw conveyor and the housing,
- bearings at the drive end for facilitating
rotation of the single or twin screw conveyor, and

- 5 an outlet for the product polymer to flow from
the housing.
4. The method of any of claims 1-3 further comprising at
least one of the following: heating the polymer-solvent
10 solution in the thermal dryer to a temperature of from
50°C to 290°C, agitating the polymer-solvent solution in
the thermal dryer, or injecting an inert stripping agent
into the thermal dryer to facilitate residuals
separation.
- 15 5. The method of any of the preceding claims further
characterized by:
- (E) feeding an initial polymer-solvent solution with a
residuals level of 70 percent by weight or greater
20 to a devolatilization reactor,
- (F) devolatilizing the initial polymer-solvent solution
in the devolatilization reactor producing a feed
polymer with a residuals level of 30 percent to 50
percent by weight, and
- 25 (G) feeding the feed polymer-solvent solution to the
thermal dryer to produce the polymer-solvent
solution.
6. The method of any of the preceding claims wherein the
30 vapor is removed by a vacuum system in fluid
communication with the thermal dryer.
7. The method of claim 6 wherein a vacuum from 10 mm Hg to
200 mm Hg (1.3 to 27 kPa) is maintained within the
35 thermal dryer by the vacuum system.
8. The method of any of the preceding claims further
characterized by:
- (E) condensing and collecting solvent from the vapor
40 removed by the vacuum system.

- 5 9. The method of any of the preceding claims wherein the product polymer is suitable for processing by a pelletizing machine and the method is further characterized by:
- 10 (E) feeding the product polymer to a pelletizing machine, and
- (F) producing pelletized product polymer with the pelletizing machine.
- 15 10. The method of any of the preceding claims wherein solvent level in the polymer product is less than 2000 parts per million.
- 20 11. The method of claim 1 wherein the polymer is selected from the group consisting of EPDM, heterogeneous polyethylene, homogeneous polyethylene, linear polyethylene, low density polyethylene, polypropylene, ethylene propylene rubber, and polystyrene.
- 25 12. The method of any of the preceding claims wherein the polymer is a terpolymer of ethylene, an alpha-olefin, and a diene, and wherein the residual diene level in the polymer product is less than 100 ppm.
- 30 13. The method of any of the preceding claims wherein the polymer has a Mooney viscosity greater than 20.
14. The method of any of the preceding claims wherein the polymer has a melt index less than 1 g/10 minutes.
- 35 15. The method of any of the preceding claims wherein the polymer-solvent solution has a residence time in the thermal dryer of less than 50 minutes.
- 40 16. The method of claim 1 wherein residuals are initially present in the polymer-solvent solution at a level of between 5 percent and 80 percent by weight, and wherein

- 5 the residuals level in the polymer product is less than
 0.2 percent by weight.
17. The method of claim 1 wherein the method is performed in
 the absence of at least one of oxygen or water.
- 10
18. The method of any of the preceding claims wherein the
 devolatilization of the polymer-solvent solution does not
 produce any significant polymer degradation.
- 15
19. A method for devolatilizing a polymer having a Mooney
 viscosity greater than 50, the method characterized by:
- (A) introducing a polymer-solvent solution into a
 thermal dryer,
- (B) treating the polymer-solvent solution in the thermal
20 dryer forming polymer product and separated solvent
 forming product polymer and separated solvent, the
 product polymer having at most 0.5 percent by weight
 solvent, and other residuals, separated solvent with
 residuals therein vaporizing in the thermal dryer
25 forming a vapor containing solvent and residuals,
 wherein the polymer-solvent solution has a residence
 time of less than 50 minutes in said dryer,
- (C) removing the vapor from the thermal dryer using a
 vacuum in fluid communication with said dryer from
30 10 mm Hg to 200 mm Hg (1.3 to 27 kPa), and
- (D) discharging product polymer with solvent removed
 therefrom from the thermal dryer.
20. A discharge system for use with a thermal dryer to dry a
35 polymer product comprising:
 a single or twin screw conveyor rotatably mounted
 in a housing, the housing having an interior into which
 product polymer flows through a housing inlet,
 a direct drive motor at a drive end of the
40 housing connected to the single or twin screw conveyor
 for rotating it,

5 dual mechanical seals at the drive end of the
housing for sealing an interface of the single or twin
screw conveyor and the housing,
 bearings at the drive end for facilitating
rotation of the single or twin screw conveyor, and
10 an outlet for the product polymer to flow from
the housing.

FIG.1

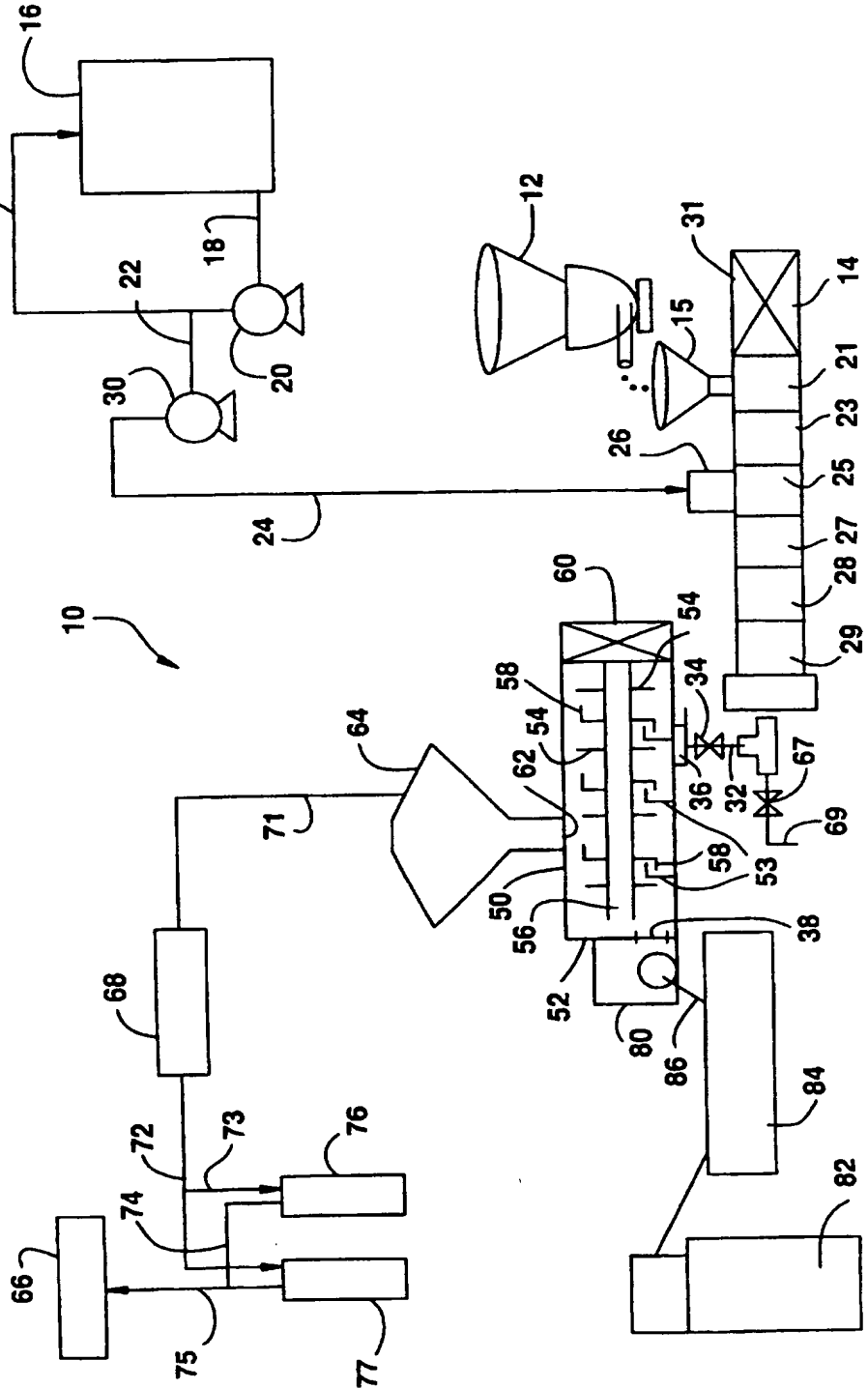


FIG.2

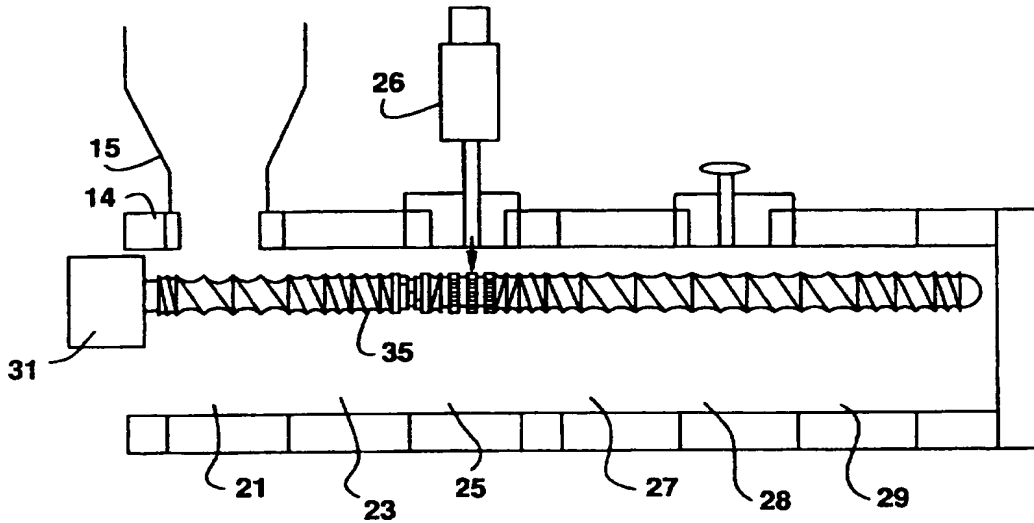
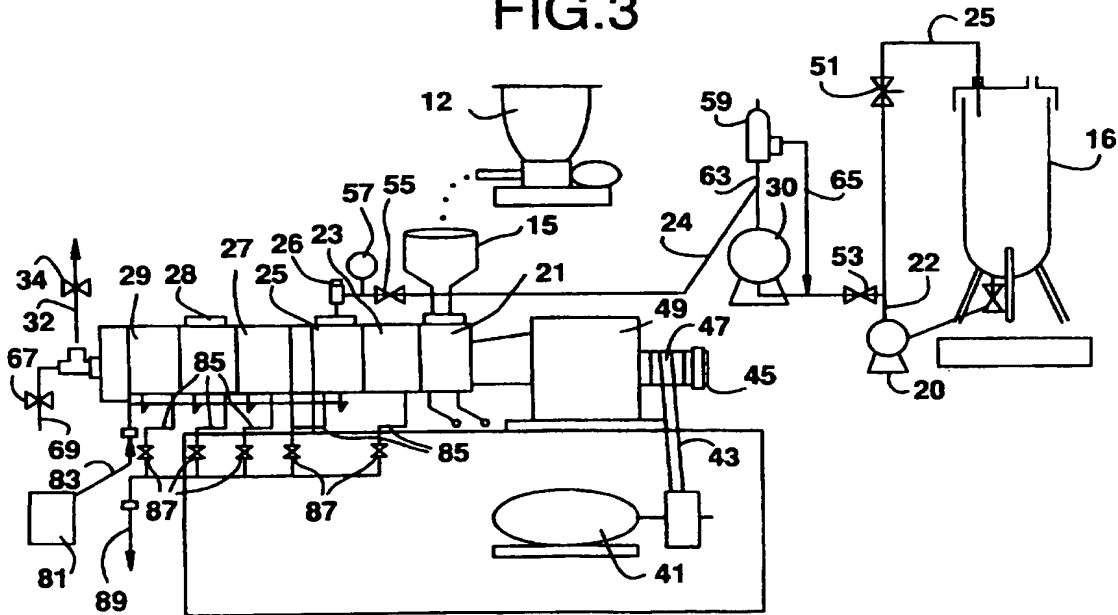
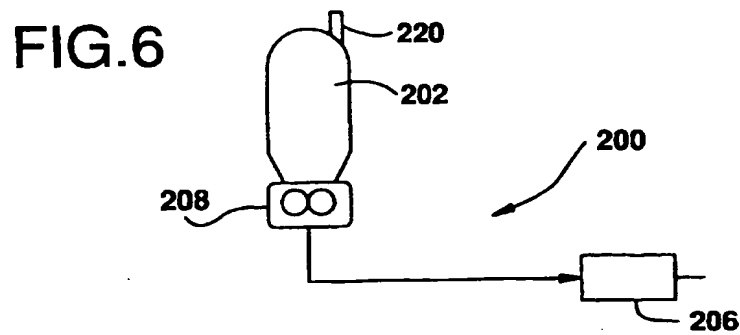
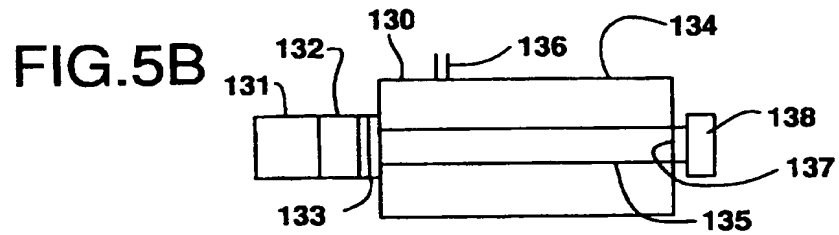
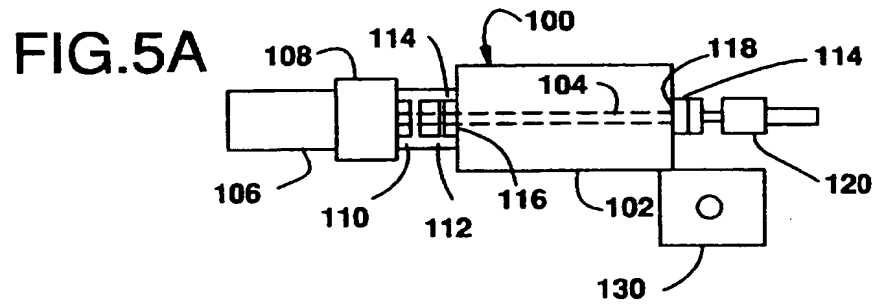
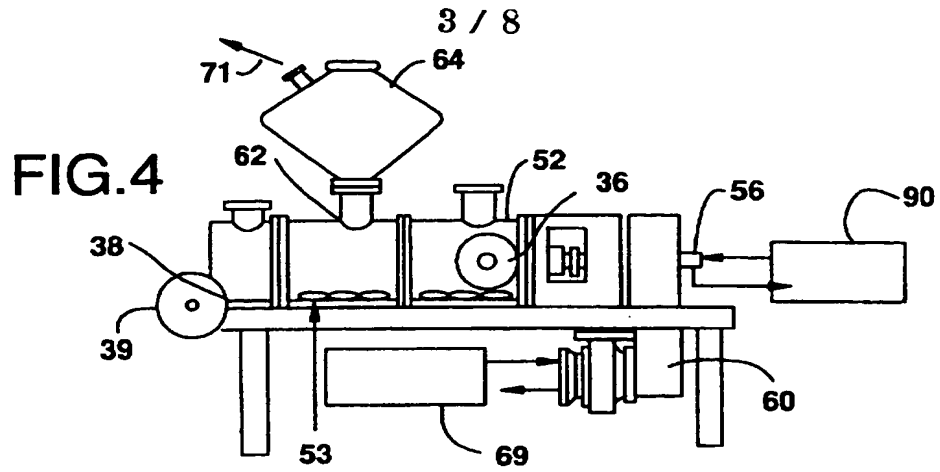


FIG.3





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FIG.7

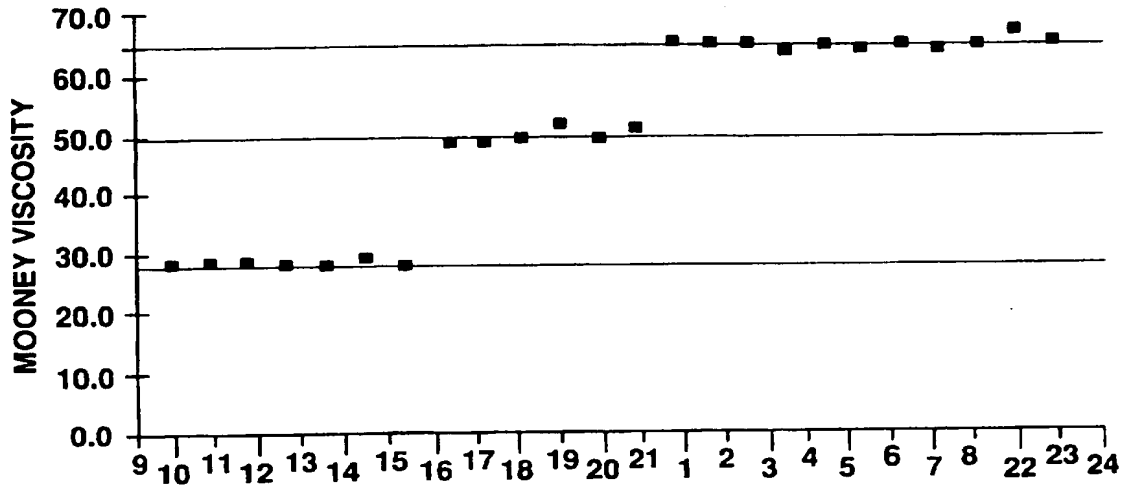


FIG.8

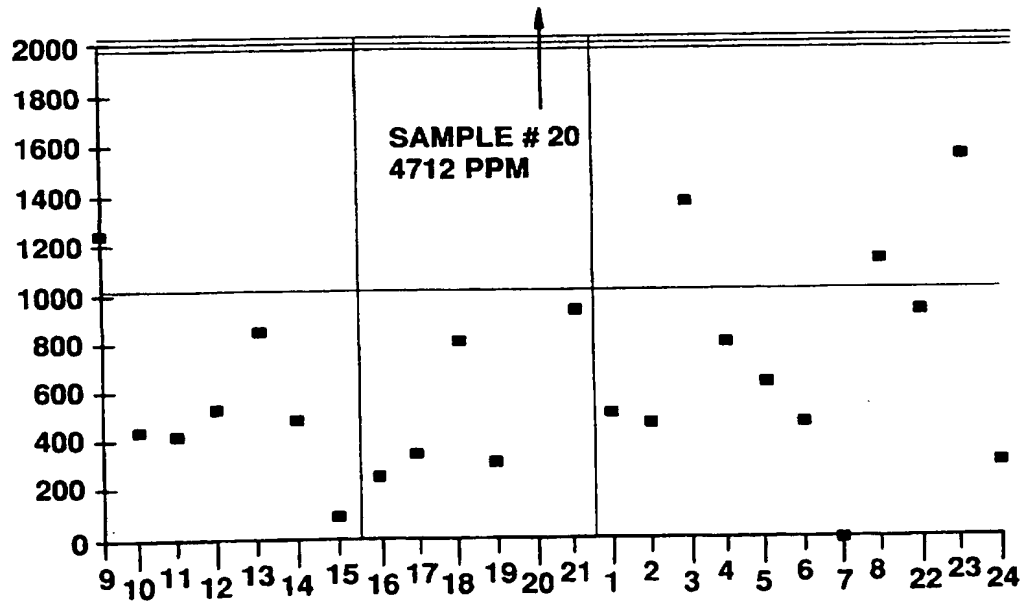


FIG.9

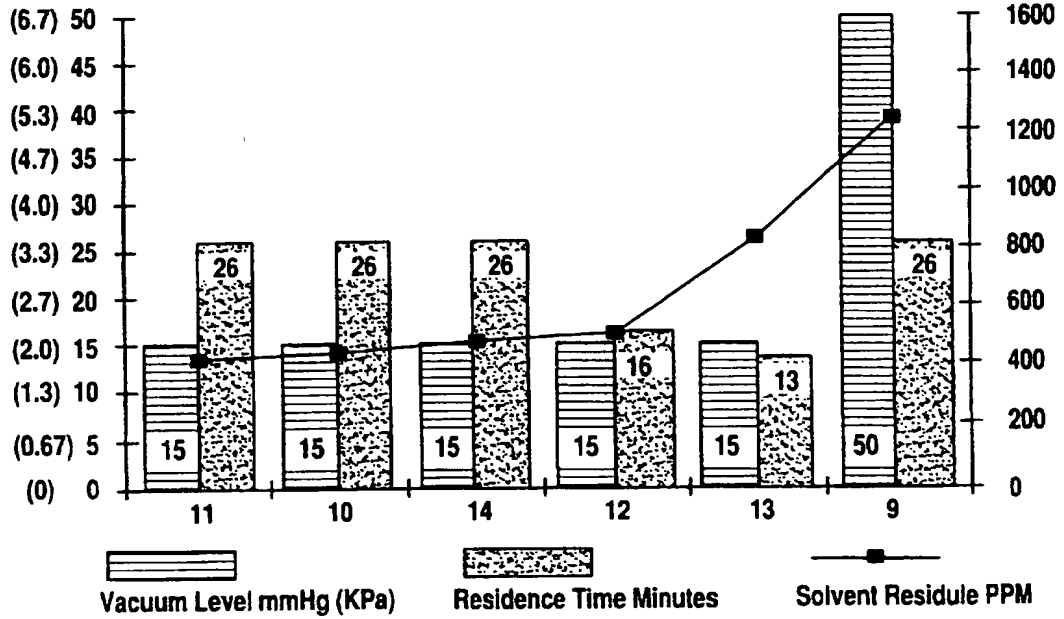


FIG.10

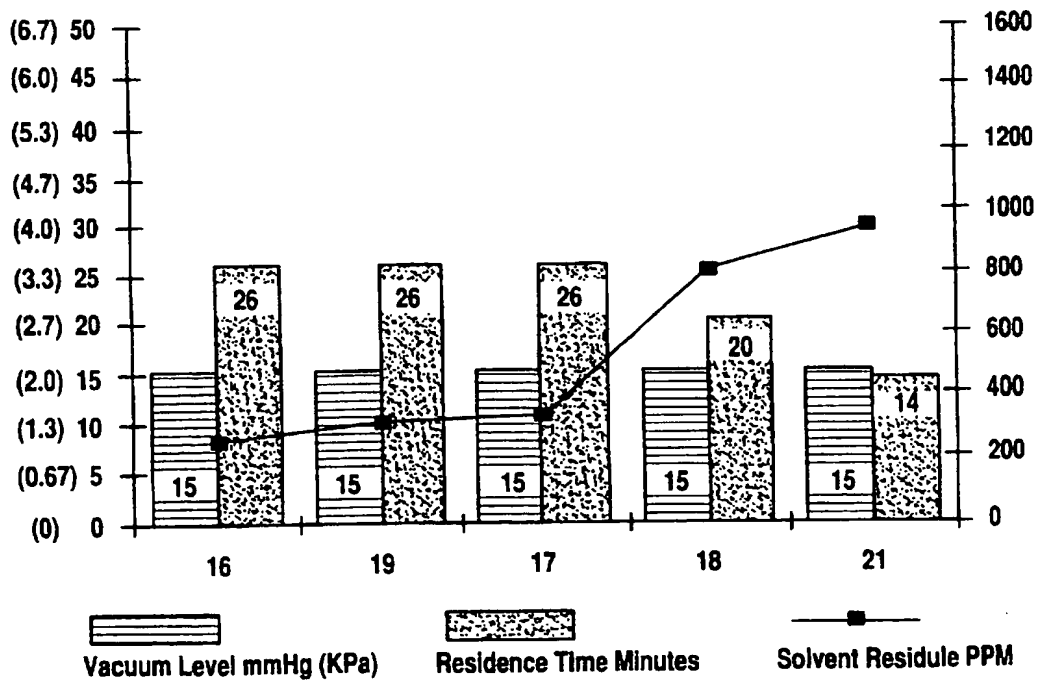


FIG.11

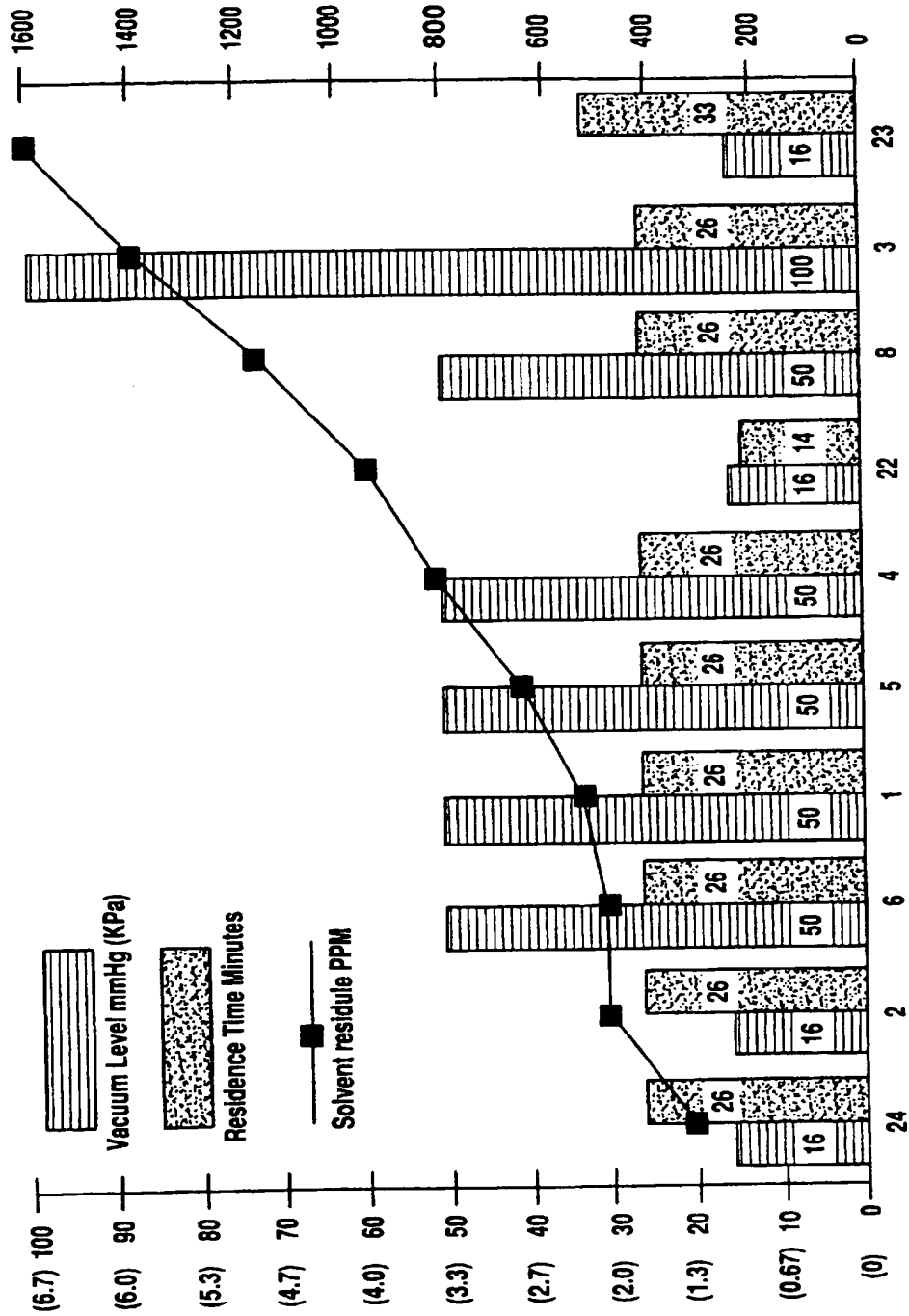


FIG.12

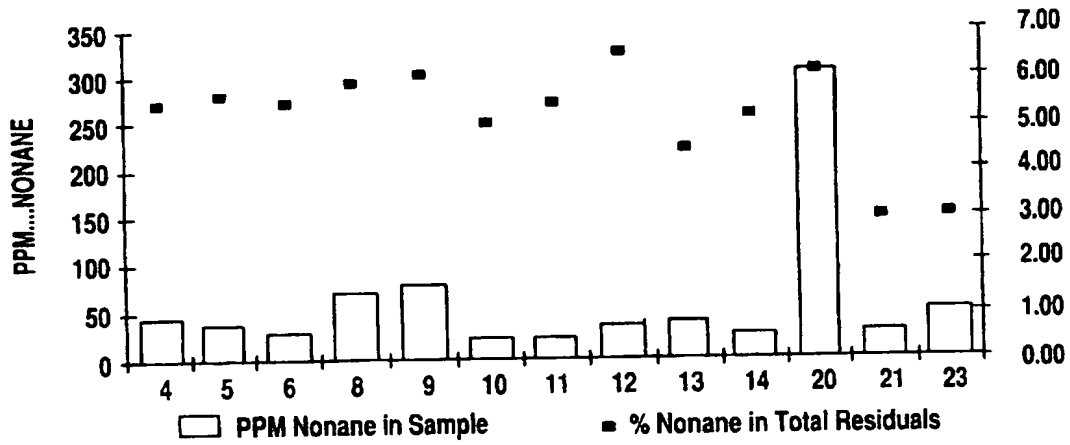


FIG.13

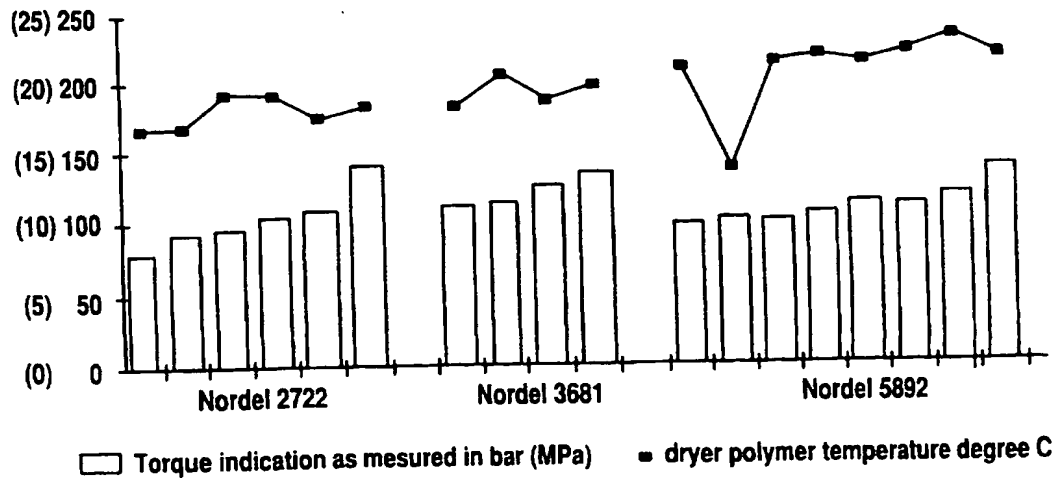


FIG.14

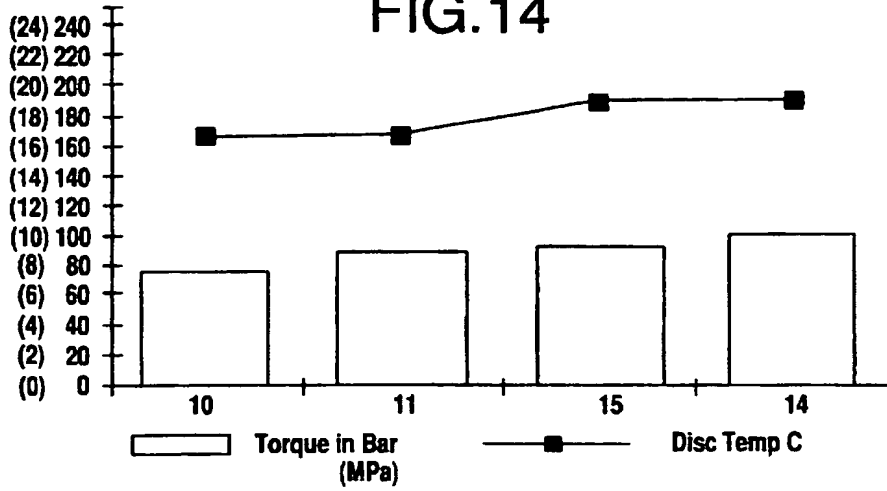


FIG.15

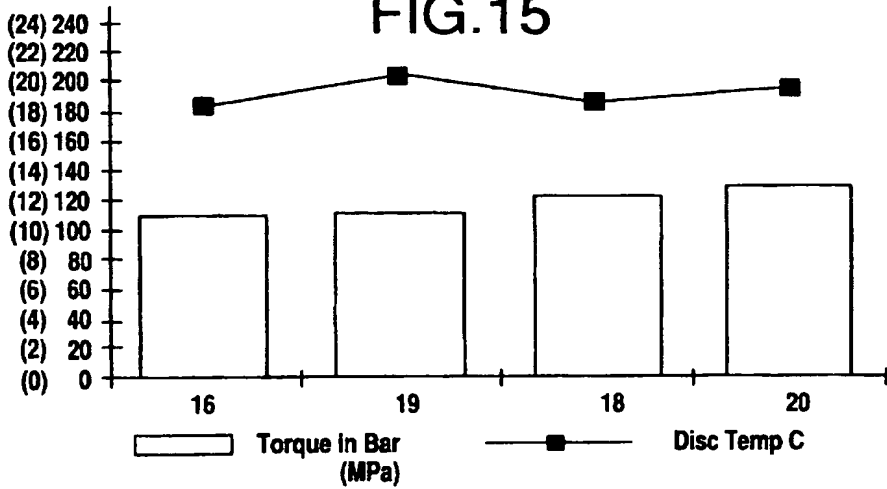
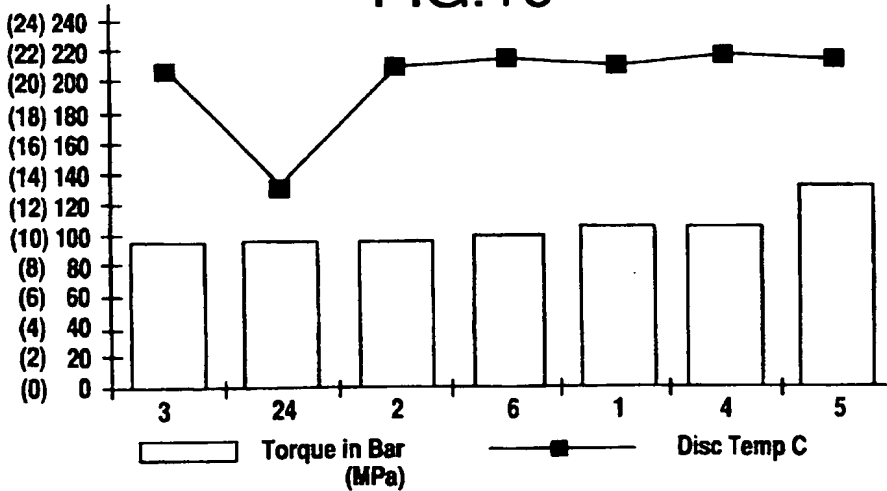


FIG.16

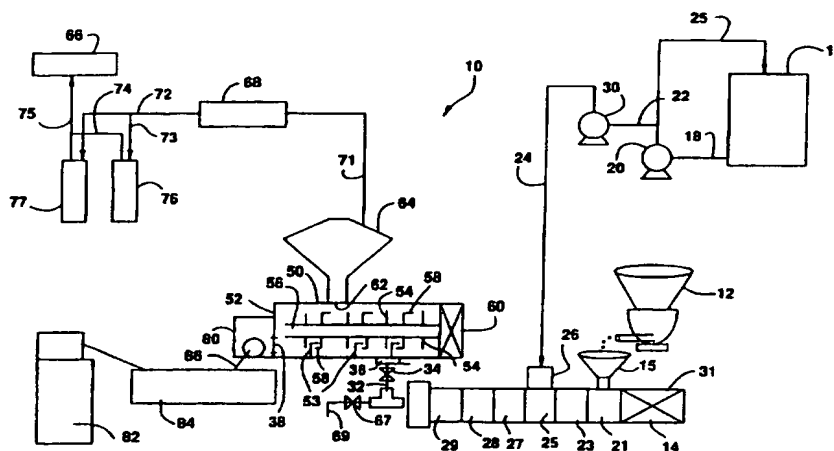




INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C08F 6/10, B01D 1/22	A3	(11) International Publication Number: WO 98/02467 (43) International Publication Date: 22 January 1998 (22.01.98)
<p>(21) International Application Number: PCT/US97/10102</p> <p>(22) International Filing Date: 8 July 1997 (08.07.97)</p> <p>(30) Priority Data: 60/021,678 12 July 1996 (12.07.96) US</p> <p>(71) Applicants (for all designated States except US): THE DOW CHEMICAL COMPANY [US/US]; 2030 Dow Center, Midland, MI 48674 (US). E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US).</p> <p>(72) Inventors; and (75) Inventors/Applicants (for US only): ABEL, Richard, C., Jr. [US/US]; 336 Timbercreek Drive, Lake Jackson, TX 77566 (US). COMBS, Robert, B. [US/US]; 93 Rosewood Drive, Lake Jackson, TX 77566 (US). ROWLAND, Michael, E. [US/US]; 402 Daffodil, Lake Jackson, TX 77566 (US). SOAPE, Jerry, W. [US/US]; 7415 Pebble Beach, Beaumont, TX 77707 (US). SMITH, Stanley, W. [US/US]; 124 Tulip Trail, Lake Jackson, TX 77566 (US).</p> <p>(74) Agents: WARRICK, Noreen, D. et al.; Patent Dept., B-1211, 2301 Brazosport Boulevard, Freeport, TX 77541 (US).</p>	<p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p>Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p> <p>(88) Date of publication of the international search report: 19 February 1998 (19.02.98)</p>	

(54) Title: POLYMER RECOVERY



(57) Abstract

Methods for devolatilizing polymer solutions have been invented which include, in certain aspects, dissolving a viscous polymer in a solvent forming a polymer-solvent solution, introducing the polymer-solvent solution into a thermal dryer, heating or cooling the polymer-solvent solution in the thermal dryer forming product polymer with solvent removed and separated solvent (which may include other residuals), the separated solvent with other residuals if present vaporizing in the thermal dryer forming a vapor, removing the vapor from the thermal dryer, and discharging product polymer from the thermal dryer.

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INTERNATIONAL SEARCH REPORT

In national application No.
PCT/US 97/10102

A. CLASSIFICATION OF SUBJECT MATTER

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According to International Patent Classification (IPC) or to both national classification and IPC

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0102122 A1 (STAMICARBON B.V.), 7 March 1984 (07.03.84), page 3, line 19 - line 29, abstract, example, claims --	1-3, 5-6, 10, 19-20
X	EP 0226204 A2 (GENERAL ELECTRIC COMPANY), 24 June 1987 (24.06.87), column 4, line 38 - line 45, claims 1-18, abstract --	1-2, 4-6, 11-12, 19
X	EP 0369708 A1 (POLYSAR FINANCIAL SERVICES S.A.), 23 May 1990 (23.05.90), abstract, claims --	1, 5

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INTERNATIONAL SEARCH REPORT

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Information on patent family members

SA 59743

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International application No.
PCT/US 97/10102

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